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Indian Standard
SPECIFICATION FOR ANILINE, TECHNICAL
(First Revision)

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Indian Standard
SPECIFICATION FOR ANILINE, TECHNICAL
(First Revision)

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Indian Standard
SPECIFICATION FOR ANILINE, TECHNICAL
(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 12 June 1973, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Chemical Division Council.

0.2 Aniline (C_6H_7N) finds extensive use in the dye industry. It is the starting material for many azo dyes. It is used in the manufacture of other intermediates as acetanilide, acetoacetanilide, aminoazobenene, methyl aniline, diethyl aniline, dimethyl aniline, and many others.

0.3 This standard first issued in 1964, is now being revised with a view to making it appropriate to the current needs of the industry. Modifications of almost all the requirements have been necessitated and maximum moisture content has been specified. Newer techniques of analysis, such as polarography has been used for the determination of nitrobenzene in aniline.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for aniline, technical.

2. REQUIREMENTS

2.1 Description — The material shall be a transparent oily liquid colourless to pale yellow when freshly distilled having a strong characteristic odour. It darkens on storage.

2.2 The material shall also comply with the requirements given in Table 1.

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*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR ANILINE, TECHNICAL

(*Clause* 2.2)

Sl No.	Characteristic	Requirement	Methods of Test, Ref to	
			Appendix	Cl No. in IS: 5299-1969*
(1)	(2)	(3)	(4)	(5)
i)	Relative density at 27°C/27°C	1·018 to 1·019	—	4
ii)	Crystallizing point, *Min*	−6·4°C	—	7
iii)	Distillation range	5 to 95 ml shall distil within the range of 1°C including 184°C	—	6
iv)	Assay, percent by mass, *Min*	99	A	—
v)	Nitrobenzene content, ppm, *Max*	50	B	—
vi)	Moisture content, percent by mass, *Max*	0·3	—	9.1

*Methods of sampling and tests for dye intermediates.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in suitable drums conforming to IS : 2552-1970*.

3.2 Marking — Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name of the manufacturer;
c) Lot or batch number;
d) Tare, net and gross mass; and
e) POISON [red label, printed on white background conforming to IS : 1260 (Part I) - 1973†].

3.2.1 Each container shall, in addition, bear the minimum cautionary notice worded as under:

KEEP WELL CLOSED AND PROTECTED FROM LIGHT.
TOXIC. AVOID CONTACT WITH SKIN

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*Specification for steel drums (galvanized and ungalvanized) (*first revision*).

†Pictorial markings for handling and labelling of goods: Part I Dangerous goods (*first revision*).

3.2.2 Each container may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in **3** of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Tests for the determination of crystallizing point and assay shall be conducted on each of the individual samples.

4.2.2 Tests for the determination of all other characteristics given in Table 1 shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirements of crystallizing point and assay by bromide-bromate method, if each of the individual test results satisfies the relevant requirements given in Table 1.

4.3.2 *For Composite Samples* — For declaring the conformity of a lot to the requirements of all the other characteristics tested on the composite sample, the test result for each of the characteristics shall satisfy the relevant requirement given in Table 1.

5. QUALITY OF REAGENTS

5.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS: 1070-1960†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

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*Methods of sampling and test for dye intermediates.

†Specification for water, distilled quality (*revised*).

APPENDIX A

(*Table* 1)

ASSAY

A-1. GENERAL

A-1.1 Outline of the Method — The material is titrated with previously standardized bromide-bromate solution. The following reaction takes place:

$$NaBrO_3 + 5\ NaBr + 6\ HCl \rightarrow 3\ Br_2 + 6\ NaCl + 3\ H_2O$$

$$C_6H_5NH_2 + 3\ Br_2 (\text{Aniline}) \rightarrow C_6H_2Br_3NH_2 + 3\ HBr.$$

A-2. REAGENTS

A-2.1 Standard Bromide-Bromate Solution — 0·1 N. Dissolve 10 g of sodium bromide and 2·75 g of sodium bromate in 1 000 ml water. Pipette out a 100-ml aliquot of this solution into a 1 000-ml iodine flask. Add 100 ml of water and 10 g of potassium iodide, and shake to dissolve. Then add 15 ml of concentrated hydrochloric acid rapidly and stopper the flask. Shake vigorously. Allow to stand for 5 minutes. Titrate with sodium thiosulphate solution 0·1 N using 2 ml of starch solution as an indicator. Disappearance of blue colour indicates the end point. Calculate the normality as follows:

$$\text{Normality of bromide-bromate solution} = \frac{\text{Titre reading} \times \text{Normality of } Na_2S_2O_3 \text{ solution}}{100}$$

NOTE — Same solution shall not be used after three months.

A-2.2 Potassium Iodide Solution — approximately 10 percent (*m*/*v*).

A-2.3 Sodium Thiosulphate Solution — 0·2 N.

A-2.4 Starch Indicator — Weigh 1 g of soluble starch and paste it with a few drops of water. Transfer this paste to 100 ml of boiling water.

A-2.5 Concentrated Hydrochloric Acid — conforming to IS: 265-1962*.

A-3. PROCEDURE

A-3.1 Weigh accurately about 0·45 g (about 1/200 mole) of the sample. Dissolve it in 1 ml of glacial acetic acid and a small amount of water. Transfer it into a 250-ml volumetric flask and dilute to the mark with distilled water.

A-3.1.1 Take 150 ml of water and 35 ml of 0·1 N bromide-bromate solution into a 500-ml iodine flask. After adding accurately 25 ml of sample solution into it, add 5 ml of hydrochloric acid quickly. After applying stopper immediately shake it to mix completely. Leave it for 10 minutes in a dark place. After making the solution absorb bromine vapour, add 10 ml of potassium iodide solution. Then titrate the liberated

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*Specification for hydrochloric acid (*revised*).

free iodine with 0·2 N sodium thiosulphate solution. When the titration is advanced, and the solution shows slightly light yellow colour, add 10 ml of starch solution. The end point is reached when the blue colour disappears. The titration shall be conducted at 20 to 25°C. Similarly make a blank determination on 35 ml of 0·1 N bromide-bromate solution.

A-4. CALCULATION

A-4.1 Purity, percent $= \frac{(B-C)\ N \times 93{\cdot}13 \times 250}{60{\cdot}000 \times M \times 25} \times 100$

where

B = volume in ml of sodium thiosulphate solution used with blank,
C = volume in ml of sodium thiosulphate solution used with sample,
N = normality of sodium thiosulphate solution, and
M = mass in g of the sample taken for the test.

APPENDIX B

(*Table* 1)

DETERMINATION OF NITROBENZENE CONTENT

B-1. PREPARATION OF CALIBRATION CURVE

B-1.1 Take 1·000 g nitrobenzene of special reagent grade in a 100-ml volumetric flask and make up the volume with aniline of such a grade that the wavepeak is zero at −0·4 V of half-wave electric potential. This will be *solution A*.

B-1.2 Take 20 ml of *solution A* in a 100-ml volumetric flask and make up the volume with aniline whose wavepeak is zero at −0·4 V half-wave potential. Let this be *solution B*.

B-1.3 Mix 4, 6, 8 and 10 ml of *solution B* with aniline of quality mentioned in **B-1.1** in 200-ml volumetric flask and make up the volume. (These standard solutions contain 0·002, 0·004, 0·006 and 0·008 percent of nitrobenzene, respectively.)

B-1.3.1 Polarograph the above solutions as in the case of sample and prepare the calibration curve from the waveheights obtained.

B-2. PROCEDURE

B-2.1 Take suitable amount of sample in the polarographic cell containing mercury and add 1 ml of concentrated hydrochloric acid. Pass nitrogen immediately. Put the cell in a thermostatic bath of 30°C for 20 minutes with the nitrogen gas flow. Polarograph this solution over an applied voltage range of 0 and $-1{\cdot}0$ V using suitable sensitivity of the galvanometer having a medium damping. The apparent half-wave potential of nitrobenzene is at $-0{\cdot}4$ V. From the waveheight obtained, the percentage is determined from the calibration curve.

B-2.1.1 *Preparation of Waveheight Diagram: (Using a D.C. Polarograph with Recorder)* — As shown in Fig. 1 draw lines *AA′* and *BB′* by extending straight lines of flat portion of the wave through the middle points of the small amplitudes of polarogram, and extend the ascending part of the wave to draw a line *CC′*. Through the points of intersection *O* and *P*, draw straight lines parallel to the horizontal axis. Take the vertical distance between *DD′* and *EE′* as waveheight.

Draw a straight line *FF′* parallel to the horizontal axis via middle point of the said waveheight. The point of intersection *M* on line *CC′* is the half-wave point and its electric potential is the half-wave electric potential.

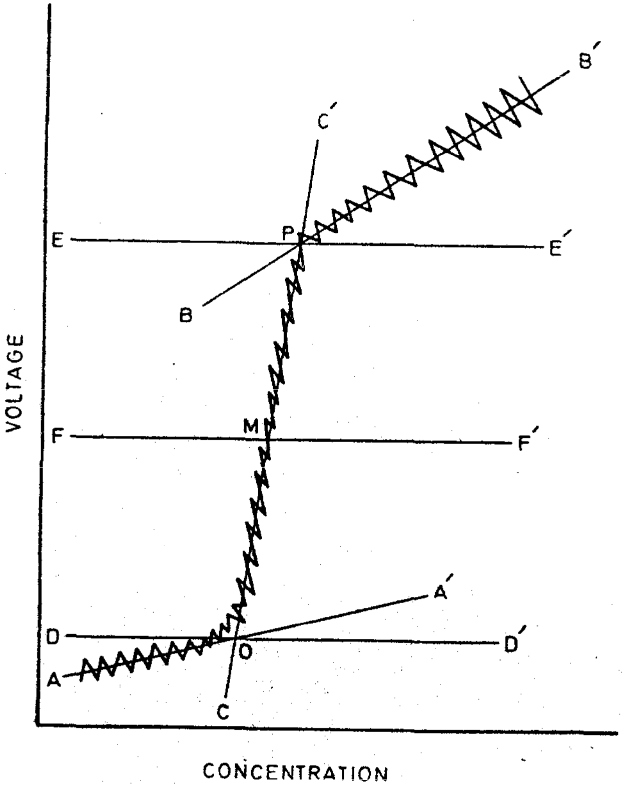

FIG. 1 POLAROGRAPHIC CURVE OF DETERMINATION OF NITROBENZENE

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbol*	*Conversion*
Force	newton	N	1 N = 1 kg.1 m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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